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ORGANIC COMPOUNDS IN PULP MILL LAGOON DISCHARGES

Bjorn F. Hrutfiord, et al

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16. ABSTRACT

Organic compounds entering and leaving kraft mills aerated lagoons have been identified and determined quantitatively. The compounds found were terpenes and related low B.P. materials, resin and fatty acids, phenols and sugar acids. The terpenes, resin and fatty acids are similar to those present in the wood specie being pulped. Some terpenes, phenols and sugar acids are produced during the pulping reactions. About 8 ppm total terpenes were found in the lagoon influent and 1 ppm or less were in the effluent. α-Terpineol was the major compound entering the lagoon and camphor the main terpene in the effluent. The total resin acid concentration entering the lagoon was 3.2 ppm with 0.6 ppm leaving. Fatty acids were lower both entering and leaving the lagoon. Sugar acids were found at about 100 ppm total entering, these were usually completely eliminated in the lagoon. Control of terpenes can be done by in-process steam stripping and the other compounds can be partially controlled by in-plant spill containment.

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ORGANIC COMPOUNDS IN PULP MILL LAGOON DISCHARGES

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ABSTRACT

This report presents information obtained in a study of the non-polymeric organic compounds entering and leaving kraft pulp mill aerated lagoons. The studies were carried out on an unbleached liner board mill pulping mostly a mixture of ~75% Douglas fir and 25% ponderosa pine, and on a specialty kraft mill that produces bleached pulp from varied wood mixes including western hemlock, western red cedar and red alder, along with Douglas fir. Both mills operate aerated lagoons of 5-8 day retention time.

Compound identification and quantitative analysis were done by classifying the compounds in three groups: terpenes and related low-boiling water insoluble; higher molecular weight acids and neutral compounds; and water soluble polysaccharide degradation products.

A number of monoterpenes were identified and quantitated. An average of about 8 ppm total terpenes was found entering the lagoon, and about 1 ppm or less was found exiting in the lagoon effluent. α-Terpineol was the terpene in largest concentration in the influent but camphor was the main compound in the effluent. Compounds present are in the pulpwood or are formed in the digester and their presence in the lagoon influent is due to a combination of physical properties. Control in-plant by steam stripping is possible.

Resin and fatty acids were identified in both the lagoon influent and effluent. Total resin acid content in the influent was 3.2 ppm while the effluent contained 0.6 ppm. Fatty acids were lower in concentration and were almost completely eliminated in the lagoon.

Water soluble organic compounds identified were mostly acids from formic up to C_6 acids related to sugars. These compounds appear in the lagoon influent in about 100 ppm total, and are lowered very effectively by the lagoon treatment, except for the acids like acetic which are metabolic products in the lagoon. There is no need for in-plant control of these compounds.

This report was submitted in fulfillment of EPA Grant No. 802084 by the University of Washington, Seattle, under the sponsorship of the Environmental Protection Agency. Work was completed as of February 1973.

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SECTION I

CONCLUSIONS

Organic compounds entering and leaving kraft pulp mill aerated lagoons have been identified and determined quantitatively. The compounds found were terpenes and related low B.P. materials, resin and fatty acids, phenols and sugar acids. The terpenes, resin and fatty acids are similar to those present in the wood specie being pulped. Some terpenes, phenols and sugar acids are produced during the pulping reactions. About 8 ppm total terpenes were found in the lagoon influent and 1 ppm or less were in the effluent. α-Terpineol was the major compound entering the lagoon and camphor the main terpene in the effluent. The total resin acid concentration entering the lagoon averaged 3.2 ppm with 0.6 ppm leaving. Fatty acids averaged 1.5 ppm entering the lagoon and 0.3 ppm leaving. Saccharinic acids were the main compounds found in lagoon influent - these averaged 124 ppm and 4 ppm was found leaving. Other small acids such as acetic and formic entered the lagoon in about 70 ppm total, the average leaving was about 56 ppm. These values remained high because sometimes a net production of formic acid occurred and several of these acids are normal metabolic products produced in this kind of wastewater treatment.

SECTION II

RECOMMENDATIONS

Further studies of this type are needed to define the nature of the organic materials being discharged from other parts of the pulp and paper industry.

The methods used here are useful in determining how any wastewater treatment facility is performing, in particular how the systems being utilized for treatment of mixed industrial and domestic sewage are performing. Determining % BOD reduction and other general parameters is no longer an adequate definition of performance.

This study and related ones have fairly well defined how the secondary treatment systems used by pulp mills operate. Any further improvements in effluent water quality should be brought about by in-plant control procedures rather than additional external treatment facilities.

SECTION III

INTRODUCTION

The nature of effluent from pulp mills, particularly sulfite mills, has been studied by numerous workers. The nature of kraft pulp mill effluent is not as well known, mainly because these mills have always practiced chemical recovery and consequently have had less discharge. With recent emphasis on the overall nature of organic contaminants in waters of the United States, the question of what is being discharged by pulp mills using accepted waste water treatment needs to be answered.

The size of the pulp and paper industry is very large - about 120 million tons of pulp are now produced annually in the U.S. and at present about 36,000 gallons of water is used per ton of pulp produced. The pollution load carried by this water discharged from unbleached kraft mills is 43 lbs BOD_5 and 37 lbs suspended solids per ton of product (1).

Most of these kraft mills practice primary and secondary treatment that removes 55 to 95% of the suspended solids and about 40 to 50% of the BOD_5 from the discharged water.

Aerated lagoons in use in the Pacific Northwest today usually remove about 85 to 90% of the BOD_5. Although this is a satisfactory accomplishment, BOD is concerned only with readily metabolized material and there are several kinds of organic compounds present in kraft mill effluents that are resistant to decomposition by microorganisms.

Review of the literature indicates that kraft mill effluents may contain a very wide variety of compounds; alcohols such as methanol, acids including formic, metasaccharinic, as well as fatty and resin acids, terpenes like α-terpineol and phenolic compounds such as guaiacol. Many others are undoubtedly present. Of these, at least the terpenoid compounds are resistant to microbial action and also they may be toxic to marine organisms. There is very little information of the fate of such compounds in aerated lagoons.

Keith, in several reports[2-5] has shown that some monoterpenes pass through aerated lagoons with partial structural changes. Anderson has shown that sugars present in sulfite liquor are significantly reduced in concentration in an aerated lagoon (6).

With this general background, study of the organic compounds in kraft mill effluent has been initiated.

SECTION IV

OBJECTIVES

The project objectives have been defines as follows:

a) To qualitatively identify and quantitatively determine the organic compounds entering the receiving water from kraft pulp mills using aerated lagoons. The organic compounds are understood as lower molecular weight compounds such as extractives and simple phenols, as well as chlorinated, sulfur bearing and toxic compounds. Other compounds which are of low molecular weight but are polar and water soluble will be included. Higher molecular weight phenolic or lignin type compounds are not to be considered in this study.

b) To determine the effect of the aerated lagoon treatment on the organic compounds by applying the sampling and testing of wastewater both before and after the aerated lagoon. These results would be supplemented by alternate pollutant measurements such as total organic carbon.

c) To provide information on the origin of these compounds in the process with the aim of defining possible control points within the process.

The study was organized into three general areas in order to meet the above objectives. Each area was the responsibility of one graduate student. The areas of study consist of neutral compounds, acidic compounds and polar compounds.

SECTION V

DESCRIPTION OF THE SPRINGFIELD MILL AND SAMPLING SITES AT THIS MILL

A study of the influence of an areated lagoon on the organic compounds in kraft mill effluent was initiated with samples from the Weyerhaeuser Company kraft mill located at Springfield, Oregon. This mill typifies an unbleached kraft mill. The mill produces 1250 air dry tons of pulp per day of unbleached linerboard from a wood mix of about 80% Douglas fir and 20% Ponderosa Pine. Both batch and continuous digesters are used along with a variety of other processing units typical of a modern kraft mill.

The effluent treatment system consists of two settling ponds used for primary treatment, and a larger aerated lagoon with a retention time of seven to eight days. A schematic of the mill effluent system is shown in Figure 1. Waste water from the pulp mill, including any water from the causticizing, cooking, washing, recovery and screening areas, is fed to the settling pond at a rate of about 3500 GPM. During the first part of the study, hydraulic barker and plywood plant waste water streams were routed to the outfall via the log pond, which also received aeration. One of these streams,hydraulic barker, wastewater, was rerouted into the settling pond about midway through the study. Water from the settling pond is fed to the retention basin and about 3200 GPM of wastewater from the paper mill is also added to this basin. This includes water from the machine room, evaporators, deckers, various spills and floor drains. After a settling period, the water flows to the aerated lagoon for a seven to eight day secondary treatment. The waste water is then discharged into the McKenzie River.

The main emphasis in this study was on lagoon influent and effluent. Influent samples were collected from a sampling system located between the retention basin and the aerated lagoon, and effluent samples were collected from the lagoon outfall pipeline. The sampling sites are indicated in Figure 1 as (in) and (out). The samples were all

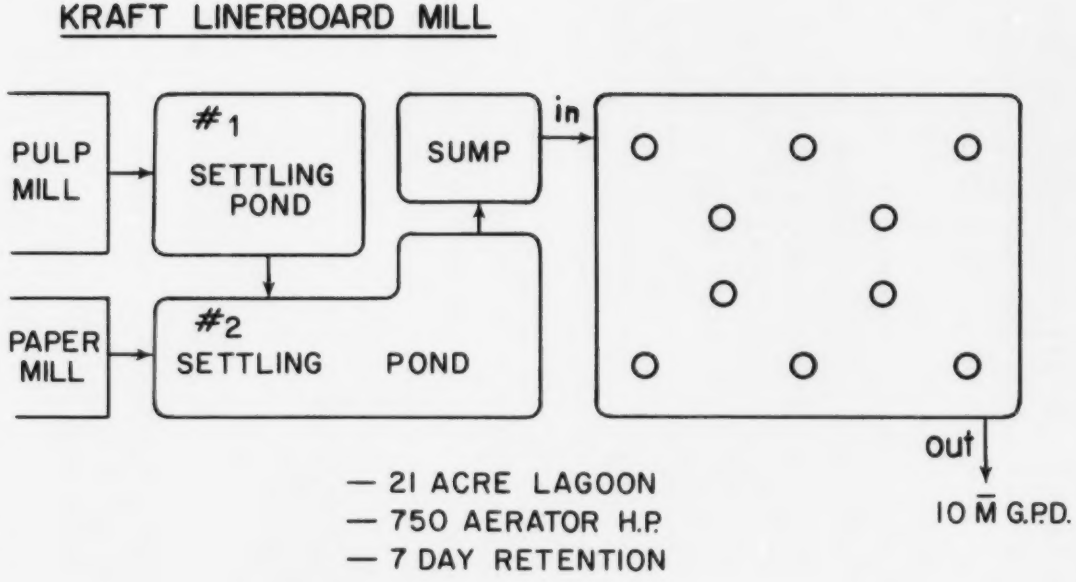

Figure 1. Schematic of the unbleached kraft mill wastewater treatment system showing sampling sites.

grab-samples and during a test sequence were taken on seven day intervals. The initial sample would be a single influent sample, followed a week later by an effluent sample and a second influent sample. This was continued for three to four weeks. The samples were shipped immediately by air to Seattle and processed, or stored at 2°C until processed.

Similar samples were collected from the Weyerhaeuser Company bleached kraft mill at Everett, Washington. This is a 500 ton mill which pulps several wood mixes, including hardwood, high percent red cedar and western hemlock-Douglas fir mixtures.

Generally, in this report, the influent and effluent data is treated in pairs, i.e. an effluent sample taken 7 days later than an influent sample is considered to represent the same material as the influent. The average retention time in the lagoon is seven days but the sample sets are not as directly related as this since the lagoon design is not a plug flow type. This should be kept in mind when making comparison of in and out concentration values.

Average data on several general water quality parameters is summarized in Table 1. The solids, BOD and COD data was provided by Weyerhaeuser and TOC determinations were made at the University of Washington. The flow rate through the lagoon during the sampling period was between 500 and 700 GPM. More detailed BOD and temperature data is presented in Table 2.

TABLE 1
GENERAL DATA ON THE SPRINGFIELD LAGOON
(ppm)

	Influent	Effluent
Solids	110-180	110-140
BOD	250-350	10-25
COD	700-900	350-500
TOC	130-140	80-100

TABLE 2

SPRINGFIELD AERATED LAGOON PERCENTAGE BOD_5 REDUCTION AND LAGOON TEMPERATURE FOR 1973

Date	Days No.	% BOD_5 Reduction	Temperature (°F)	Date	Days No.	% BOD_5 Reduction	Temperature (°F)
Jan.	4	82.0	81.3	July	186	84.9	84.5
	11	81.5	73.8		193	81.9	84.0
	18	82.4	83.4		200	89.5	85.0
	25	82.5	73.8		207	89.1	84.0
Feb.	32	82.1	73.4	Aug.	214	88.2	84.2
	39	77.6	75.0		221	90.6	84.0
	46	83.4	77.0		228	82.6	84.5
	53	90.5	76.7		235	80.6	82.0
March	60	88.0	75.4		242	87.3	79.3
	67	89.1	79.0	Sept.	249	82.1	82.0
	74	89.8	75.2		256	89.4	83.2
	81	89.0	73.4		263	87.8	83.2
	88	83.3	77.0		270	90.6	82.1
April	95	83.3	79.3	Oct.	277	87.4	82.1
	102	82.4	83.1		284	90.5	76.7
	109	83.6	79.0		291	95.7	82.3
	116	91.0	78.9		298	94.4	82.1
May	123	90.2	72.4	Nov.	305	93.5	82.2
	130	89.1	80.7		312	93.9	73.0
	137	90.0	88.2		319	94.9	75.7
	144	87.3	84.0		326	92.9	71.7
	151	88.1	82.8		333	85.2	73.0
June	158	87.3	84.0	Dec.	340	85.7	73.1
	165	86.1	81.4		347	86.4	73.5
	172	83.4	82.9		354	88.7	74.5
	179	90.6	86.0		363	89.2	70.5

SECTION VI

SEPARATION AND ANALYSIS SCHEMES

A large variety of organic compounds with widely differing properties are present in kraft mill effluent. These compounds are present in low concentrations, mostly in the few ppm range or less. Thus, their detection and isolation requires processing of adequate size samples and the use of proper equipment. In this work the aim has been to use the gas chromatograph/mass spectrometer (GC/MS) combination for identification work and to use gas or liquid chromatography for quantitative determination.

Initially a considerable amount of time and effort was spent in working out analysis schemes in which all compounds isolated were determined on the same sample by sequential separations. This proved to be impractable in terms of time involved and in particular in developing reliable quantitative data. The scheme finally used involved dividing a sample into two parts and processing these individually.

The overall separation and analysis scheme used is outlined in Figure 2. This system results in fractionating the sample into three parts which are designated as neutral compounds, acidic compounds and polar compounds, according to their chemical and physical properties.

The neutral fraction was isolated by adjusting the sample to pH 11 and extracting with hexane. The extraction conditions were studied extensively and the final version recovers greater than 90% of the compounds in this class. Quantitative determination was done by gas chromatography analysis using a polyethylene glycol column. Identifications were done by GC/MS matching with known compounds, and in a few cases by matching published spectra.

Polar and acidic compounds were isolated from the samples by passing the neutralized sample over an XAD-2 resin column, which retains the higher molecular weight non-polar acids, and collecting the column eluate, which contains the polar compounds. The polar compounds were

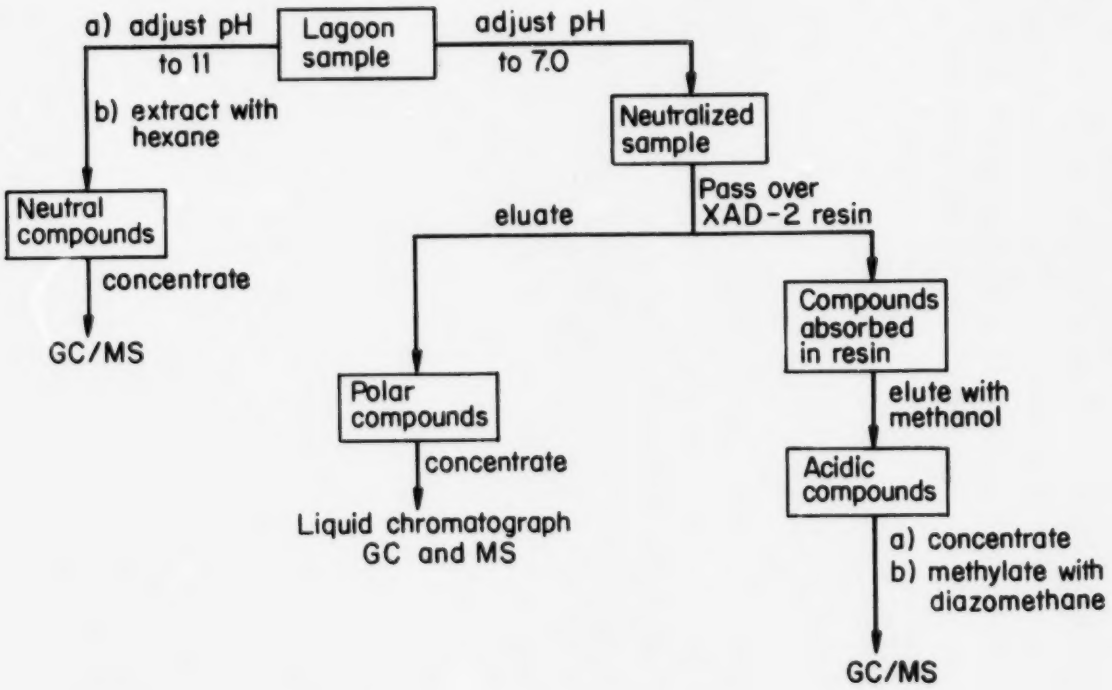

Figure 2. Lagoon sample separation scheme

determined by making the eluate alkaline, concentrating on the rotary evaporator, then buffering the sample to pH ~9.3 with 0.03 M sodium tetraborate and passing it over an anion exchange column in a liquid chromatograph equipped with a refractive index detector. Compound identifications were made by comparison with knowns, and where necessary, by collection of elution peaks, silation and determination of mass spectra using the solid probe technique. Compound identifications were made by running knowns and by comparisons with published spectra in some cases.

Acids were eluted from the resin column with methanol, taken to dryness and dissolved in ether, methylated exhaustively with diazomethane and separated by GC using a polyethylene glycol-TPA column. Identity was verified by GC/MS comparison with known compounds.

SECTION VII

NEUTRAL COMPOUNDS

Compounds discussed in this section are neutral molecules with boiling points up to about 250°C. The majority of the compounds identified in this fraction are mono- and sesquiterpene hydrocarbons and monoterpene alcohols and ketones similar to the compounds found in wood and sulfate turpentine.

The analysis system used required that the unknown compounds be separated from water and concentrated. After studying several systems, hexane extraction was adopted. Using α-terpineol in water as a model, it was demonstrated that quantitative recovery of ppm quantities could be extracted from pH 12 samples using 100 ml of redistilled hexane with a 3500 ml sample. The mixture was mechanically stirred for an hour and the extraction repeated 3 times. Combined extracts were dried and concentrated to 1 ml by careful evaporation using a Kuderna-Danish evaporator-concentrator heated on a steam bath.

Analysis was done by gas chromatography using a 100 foot Carbowax 20M support coated open tubular column in a Perkin Elmer 990 gas chromatograph. This instrument is connected with a Biemann-Watson separator to a Hitachi RMS-4 mass spectrometer.

Gas chromatographic analyses were done by injecting 0.3µl samples and programming from 90 to 150°C at 2°/minute. Results were recorded on a Hitachi Model 56 recorder and integrated with a Infotronics electronic integrator. Typical gas chromatograms from lagoon influent and effluent (for weeks 5-10 and 5-17) are shown in Figure 3. Peaks on which qualitative and quantitative data were obtained were numbered 1 through 46, as indicated on the curve.

Qualitative identifications were done by determining the mass spectra of the compounds, and wherever possible running pure knowns through the same system for verification. Literature spectra were also

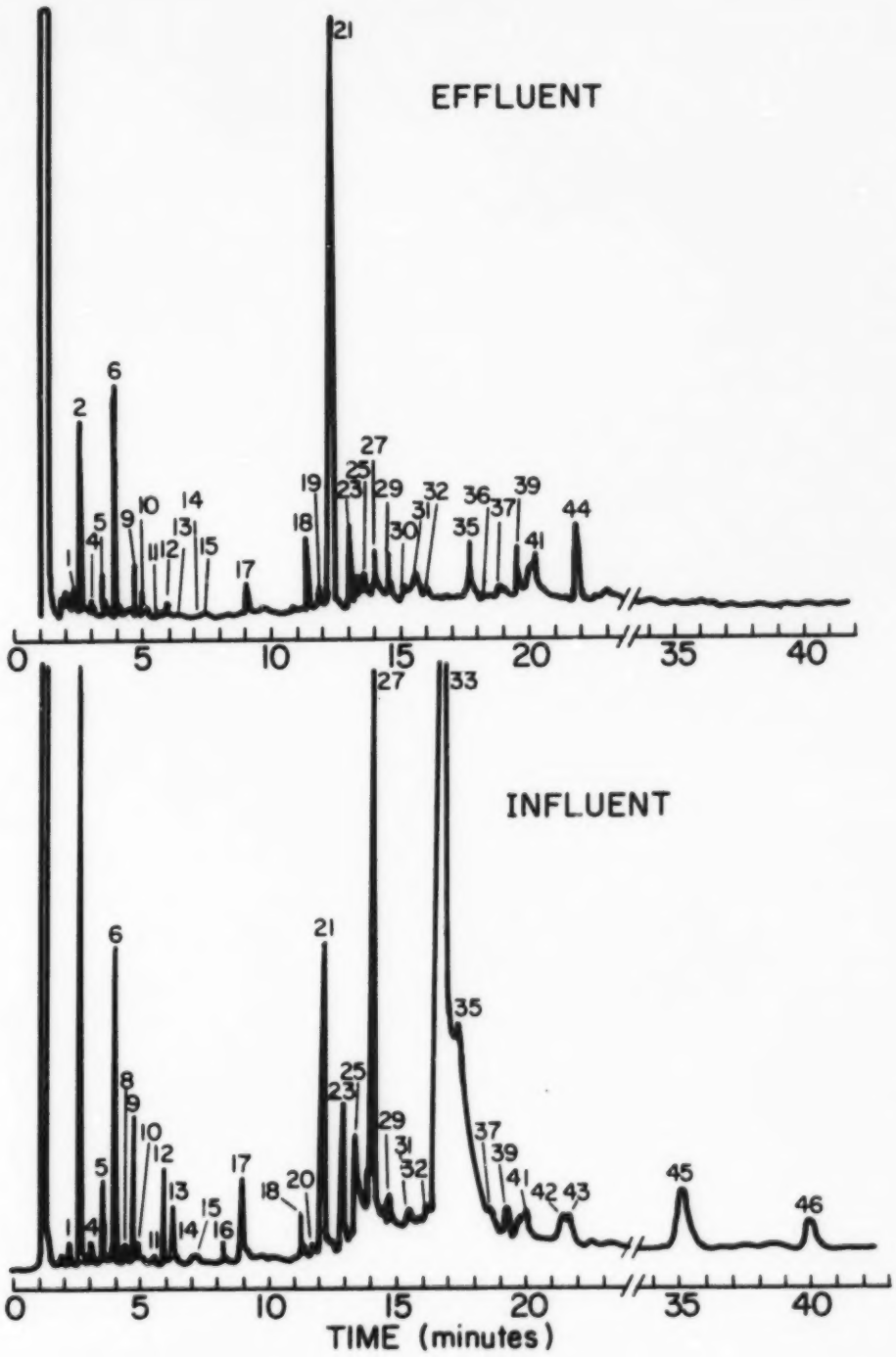

Figure 3. Gas chromatograms of neutral compounds - Springfield

used extensively. Pertinent information establishing identity of the neutral compounds is summarized in Table 3.

Quantitative data on the individual compounds was obtained from the peak areas of the gas chromatograms of the extracts. These peak areas were corrected for flame detector response by the use of compound class factors relative to limonene as 1.0. The factors used were for hydrocarbons 0.98; ketones 0.84; and alcohols 0.80. The individual quantities were then calculated from the weight of the extract (corrected for non-volatile residues) and the corrected peak areas. Finally the concentrations were calculated in ppm from the individual quantities and the sample size.

Quantitative data obtained on the Springfield lagoon is summarized in Table 4. In general, the major terpene entering the lagoon is α-terpineol accompanied by lesser amounts of other terpene alcohols, including terpinene-4-ol, and fenchyl alcohol. Trace amounts of terpene hydrocarbons and ketones are also present. The major terpene leaving the lagoon is camphor, accompanied by other hydrocarbons, alcohols and ketones with the alcohol fraction being greatly reduced. In some instances both camphor and fenchone increased in concentration in the lagoon.

The efficiency of removal of the neutral compounds by the aerated lagoon treatment is generally quite good. The average influent concentration of terpenes etc. is about 7.5 ppm and the average effluent concentration is about 0.9 ppm, giving a reduction of 88%. A summary of the % removal data is presented in Table 5. Information on BOD removal efficiency, lagoon temperature, and neutral compound concentrations in influent and effluent is summarized in Fig. 4. The trend of terpene removal showed increasing efficiency throughout the sampling period. The mill operating data showed the same trend for BOD_5 removal and these trends have a correlation coefficient of 0.75. None of the lagoon operating data explained the increasing BOD removal efficiency, in fact the lagoon temperature which normally directed influences BOD_5 removal

TABLE 3

MASS SPECTRAL DATA ON NEUTRAL COMPOUNDS IN THE SPRINGFIELD LAGOON

Peak No.	Compound Identity	Molecular Weight	M.S. Major Fragments and Comments	Lit. Ref.
1A	Solvent	84	57 43 41 71 85 56 (Hexane isomer)	(7)
1B	Unknown			
1C	Unknown			
1D	Chloroform	118	correlated with M.S. of known	
2	α-Pinene	136	✓ ✓ ✓ ✓ and published M.S.	(8)
3	Santene	122	correlated with published M.S.	(8)
4	Camphene	136	correlated with M.S. of known and published M.S.	(8)
5	Sabinene	136	✓ ✓ ✓ ✓	(8)
6	β-Pinene	136	✓ ✓ ✓ ✓	(8)
7A	Myrcene	136	✓ ✓ ✓ ✓	(8)
7B	α-Phellandrene	136	correlated with published M.S.	(8)
8	1-4 Cineole		correlated with M.S. of known	
9A	Limonene	136	correlated with M.S. of known and published M.S.	(8)
9B	Diethyl dissulfide	122	correlated with M.S. of known	
10A	1-8 Cineole		✓ ✓ ✓ ✓	
10B	β-Phellandrene	136	✓ ✓ ✓ ✓	
11	Δ^3 Carene	136	correlated with M.S. of known and published M.S.	(8)
12	p-Cymene	134	✓ ✓ ✓ ✓ ✓	(8)
13	Terpinolene	136	✓ ✓ ✓ ✓ ✓	(8)
14	Unknown	152	79 43 41 93 55 67	
15	Unknown	152	67 95 41 82 110 57	
16	Dimethyl trisulfide	126	correlated with M.S. of known	
17	Fenchone	152	correlated with M.S. of known and published M.S.	(9)
18	Unknown		10.9 81 124 50 41 55	
19	Unknown		50 41 81 120 57 55	
20	Guaiacol	124	correlated with M.S. of known	
21	Camphor	152	correlated with M.S. of known and published M.S.	(9)
22	Linalool	154	✓ ✓ ✓ ✓ ✓ M.S.	(10)

TABLE 3 (Continued)

MASS SPECTRAL DATA ON NEUTRAL COMPOUNDS IN THE SPRINGFIELD LAGOON

Peak No.	Compound Identity	Molecular Weight	M.S. Major Fragments and Comments							Lit. Ref.
			57	43	41	71	85	56		
23	Unknown	138	95	43	138	123	79	55	(camphene)	(11)
24A	Unknown		95	93	91	43	41	67		
24B	Unknown									
25	Fenchyl alcohol	154	correlated with known and published M.S.							(11)
26	Unknown	154	121	93	136	107	105	91	(trans-β-terpineol)	
27	Terpinene-4-ol	154	correlated with M.S. of known and published							(11)
28	Sesquiterpene	204	93	121	136	41	43	91		
29	Sesquiterpene	204	93	71	43	41	121	69		
30	Unknown									
31	Sesquiterpene	204	93	121	41	95	91	81		
32	Borneol	154	correlated with known and published M.S.							(11)
33	α-Terpineol	154	✓		✓		✓			(11)
34	Unknown									
35	Sesquiterpene	204	93	161	119	137	204	105		
36	Unknown									
37	Sesquiterpene	204	93	41	43	91	121	105	(longifolene	(7)
38	Unknown									
39	Sesquiterpene	204	93	68	41	136	121	43		
40	Unknown	162	119	91	43	68	39	121		
41	Anethole	148	correlated with M.S. of reference standard (p-propenyl anisole)							
42	Unknown	163	132	117	91	111	115	92		
43	Unknown									
44	Unknown	220	205	41	43	138	93	95	(cedranone)	(7)
45	Unknown	220	205	57	41	43	55	71		
46	Unknown	218	41	59	91	55	93	119		

TABLE 4

CONCENTRATION OF NEUTRAL COMPOUNDS ENTERING AND LEAVING THE SPRINGFIELD AERATED LAGOON (ppm)

GC peak [a]	Compound Identification	ABI 1-31 [b][c]	ABE 2-7	ABI 2-7	ABE 2-14	ABI 2-14	ABE 2-21
1A	*	tr [d]	tr	.04	.03	tr	tr
1B	*	.03	.03	.09	.07	.03	.04
1C	*	.04	tr	.02	.01	tr	tr
2	α-Pinene	.03	.01	.43	tr	.18	.01
4	Camphene	tr	.01	.04	tr	tr	tr
6	β-Pinene	.31	.25	1.43	tr	.04	.40
7A	Myrcene	.16	.05	.11	.06	tr	.08
7B	α-Phellandrene	.03	tr	.31	tr	.19	tr
8	1-4-Cineole	tr	tr	.01	tr	tr	tr
9A	Limonene	.03	.01	.22	.06	.03	.02
10A	1-8-Cineole	.12	.04	.16	tr	.02	.11
10B	β-Phellandrene	.03	.01	.04	.01	tr	.03
11	Δ^3-Carene						
12	p-Cymene	.07	tr	.25	tr	.12	tr
13	Terpinolene	tr	tr	.02	tr	tr	tr
17	Fenchone	.07	.18	.10	.25	.05	tr
18	*	.06	.04	.04	.08	.02	tr
21	Camphor	.40	.32	.37	.28	.20	tr
23	*	.17	.03	.15	.08	.08	tr
25	Fenchyl alcohol	.40	.01	.56	tr	.27	tr
27	Terpinene-4-ol	.72	.05	.73	.06	.37	tr
29	Sesquiterpene	.04	tr	.03	tr	.04	tr
32	Borneol	.06	tr	.11	tr	.02	tr
33	α-Terpineol	5.49	.04	5.57	.03	3.14	tr
35	Sesquiterpene				tr		
37	Sesquiterpene		.03		.07		.01
39	Sesquiterpene		.01	.07	tr		.01
42	*		.06	.04	.03	.01	tr
	TOTAL	8.26	1.19	10.92	1.23	5.02	0.71

(a) Order of Elution on K20 Column, see Figure 3
(b) Aeration Basin Influent and Effluent
(c) Sample Date
(d) tr means < 0.01 ppm

(*) unknown

TABLE 4 (Continued)

CONCENTRATION OF NEUTRAL COMPOUNDS ENTERING AND LEAVING THE SPRINGFIELD AERATED LAGOON (ppm)

GC Peak	Compound Identification	ABI 5-10	ABE 5-17	ABI 8-10	ABE 8-17	ABI 10-9	ABE 10-16
1C		.01	.05	.01	.01		
1D	Chloroform					.01	.02
2	α-Pinene	.53	.13	.06	.07	.06	.01
4	Camphene	.03	.01	tr	tr	tr	tr
5	Sabinene	.05	.04	.01	.02	.01	tr
6	β-Pinene	.22		.09	.10	.06	.04
7A	Myrcene			.01	tr		
8	1-4-Cineole			tr	tr	.01	
9A	Limonene			.02	.03	.02	.01
9B	Diethyl dissulfide	.11					
10A	1-8-Cineole	tr		.02	.02	.01	.01
12	p-Cymene	.08		.01	tr	.03	tr
13	Terpinolene	.04					
15	*				.01	tr	
16	Dimethyl trisulfide	.02		.04		.01	tr
17	Fenchone	.06		.04	.02	.05	
18	*			.04	.04	.05	
19	*			.01	.01	.01	
20	Guaiacol			.03		.07	
21	Camphor	.04	.11	.19	.35	.17	.02
23	*	.23	.06	.12	.05	.14	
24A	*	.08	tr	.01	.02		
24B	*		.12				
25	Fenchyl alcohol	.48	.10	.20	.02	.20	.02
26	*		.03		tr		
27	Terpinene-4-ol	.45		.59	.03	.52	.01
28	Sesquiterpene			.04	.03	.04	.02
29	Sesquiterpene	.04			.01	.06	.01
31	Sesquiterpene	.13			.02	.01	
32	Borneol	.02	.01	.01		.01	
33	α-Terpineol	5.12		3.72		2.95	
34	*						tr
35	Sesquiterpene			1.05	.04	.47	.04
37	Sesquiterpene			.11	tr	.44	tr
38	*					.02	.01
39	Sesquiterpene	.01	tr			tr	.01
40	*	.01	.02				
41	Anethole	.03	.06	.02	.01	.03	tr
42	*			.01	.03	.04	
43	*			.01	.06	.03	
45	*			.05	.07	.02	
46	*					.04	.01
	TOTAL	8.04	.97	6.53	1.06	5.96	.23

TABLE 5

SEASONAL EFFICIENCY OF THE SPRINGFIELD AERATED LAGOON ON NEUTRAL COMPOUNDS

Season/Date	Monoterpene Concentration influent	effluent	% Terpene Removal	% BOD Reduction
Winter				
1-31, 2-7	8.26	1.19	85.6	82.2
2-07, 2-14	10.92	1.23	88.7	83.4
2-14, 2-21	5.02	0.71	85.9	90.5
Average	8.06	1.03	86.7	85.4
Spring				
5-10, 5-17 (Aug)	8.04	0.97	87.9	90.0
Summer				
8-10, 8-17	6.53	1.06	83.7	82.6
8-20, 8-27	7.02	0.31	95.6	87.3
Average	6.86	0.68	89.6	85.0
Autumn				
10-3, 10-9	6.48	0.40	94	90.5
10-9, 10-16	5.96	0.22	96.2	95.7
Average	6.22	0.31	95.1	93.1

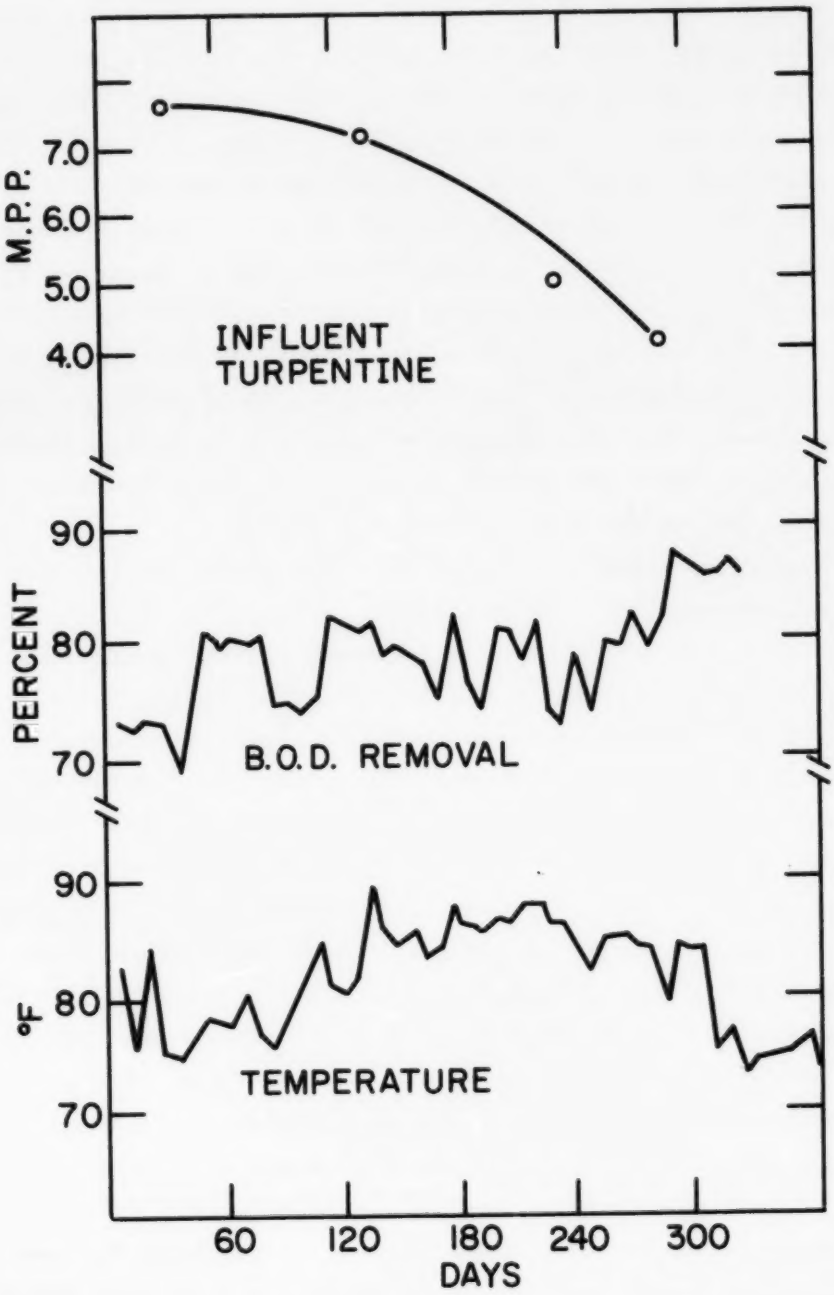

Figure 4. Temperature, BOD removal and turpentine concentration for the Springfield aerated lagoon, 1973.

showed no correlation at all (11). The influent terpene concentration, which decreased during the latter part of the sampling year, may be important here in the lagoon operating efficiency since terpenes have a well known bacterial inhibiting effect.

Lagoon samples were also obtained from a bleached kraft mill at Everett. This mill pulps several wood species including Douglas fir, western hemlock, western red cedar and red alder. These are pulped in varying ratios, with some Douglas fir present in most wood mixes. The grade of pulp produced is changed frequently and as a result the lagoon input is highly variable. Generally, sampling of lagoon influent was done 24 hours after the beginning of pulping of a given wood mix and the lagoon effluent was sampled a week later, which equals the average retention time of the lagoon.

Samples from the Everett mill were treated by procedures similar to those used for the Springfield samples.

Influent and effluent gas chromatograms from the pulping of Douglas fir, a red cedar and red aldermixture are presented in Figures 5, 6 and 7. The GC/MS identifications are given in Tables 6, 7 and 8. Concentration of the neutral compounds, in ppm, are given in Tables 9 and 10.

The GC results in Figure 5 and the compounds listed in Table 9 are from the pulping of 100% Douglas fir and can be compared to the Springfield mill results (Figure 3 and Tables 3 and 4) since the wood mix used there is about 80% Douglas fir and 20% Ponderosa pine. All sixteen monoterpenes identified at Everett were found in the Springfield lagoon and the chromatograms from the two locations are very similar. More compounds are detectable in the Everett lagoon. For the red alder and western red cedar samples, the major terpenes present in the influent were similar to the Douglas fir samples except for the tropolones identified in the influent from the cedar samples. Significantly, they did not appear in the effluent. The influent chromatogram for the alder sample was also quite similar to the Douglas fir influent though run on

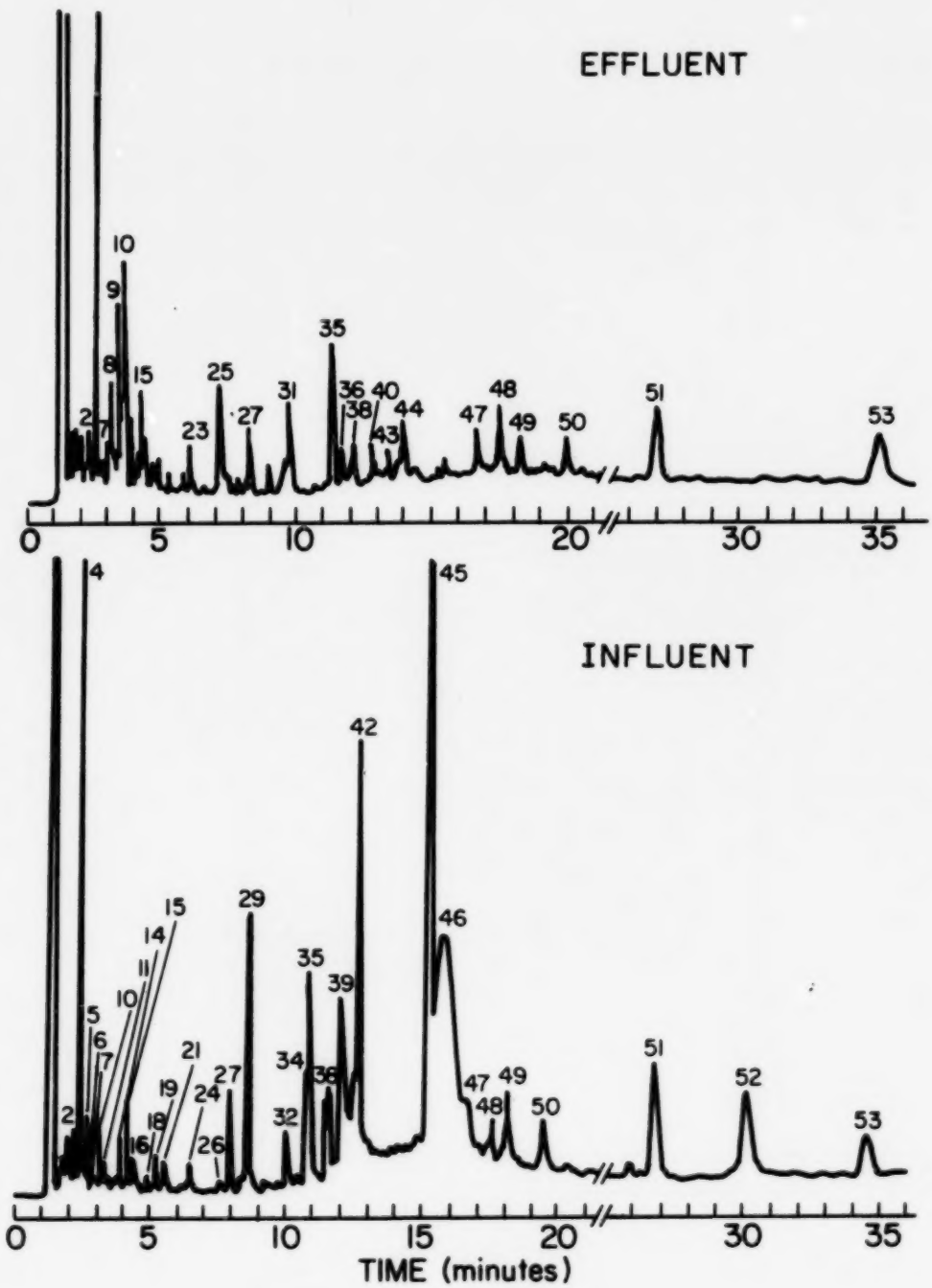

Figure 5. Chromatograms of neutral compounds from Douglas fir, Everett

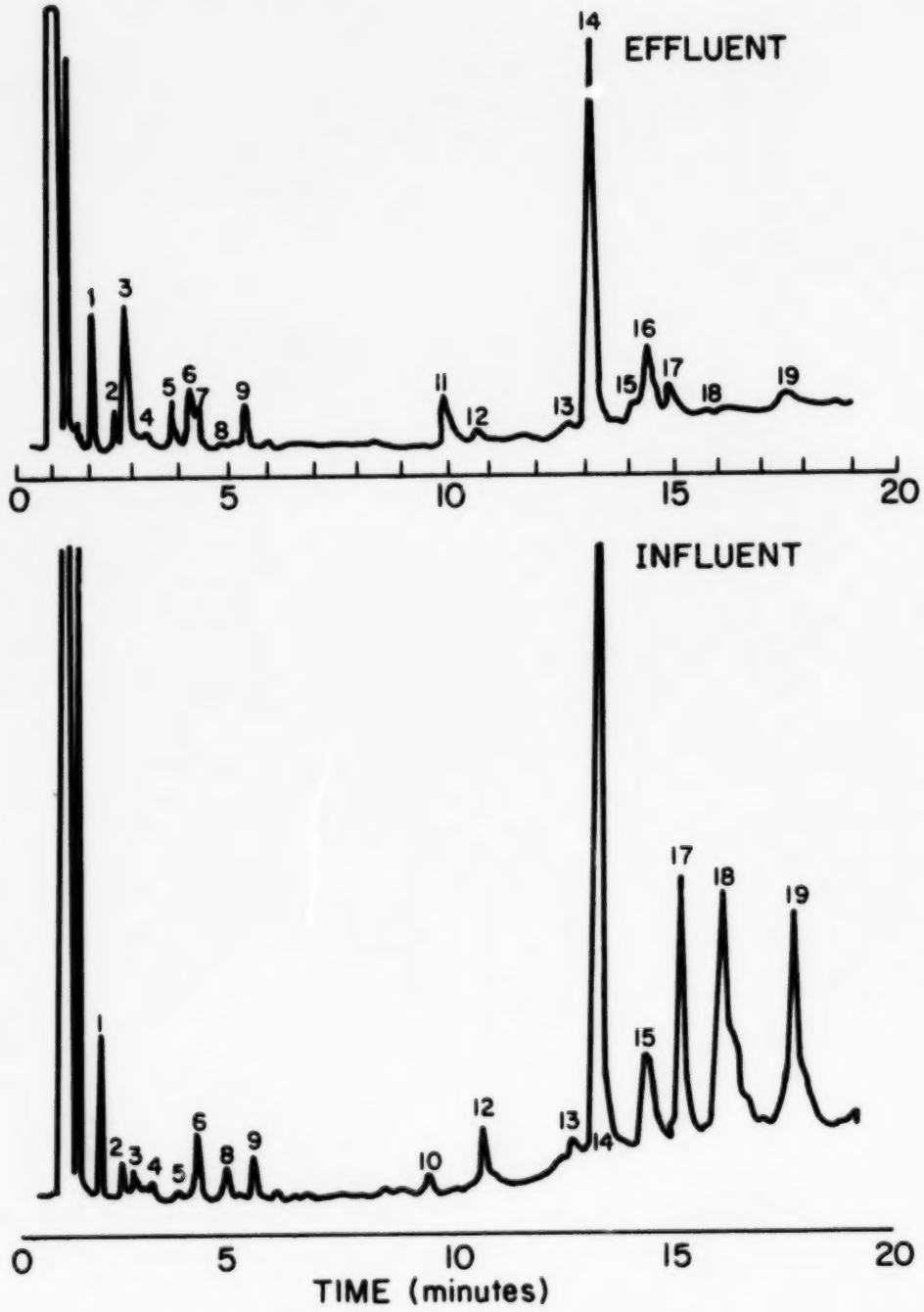

Figure 6. Chromatograms of neutral compounds from western red cedar, Everett

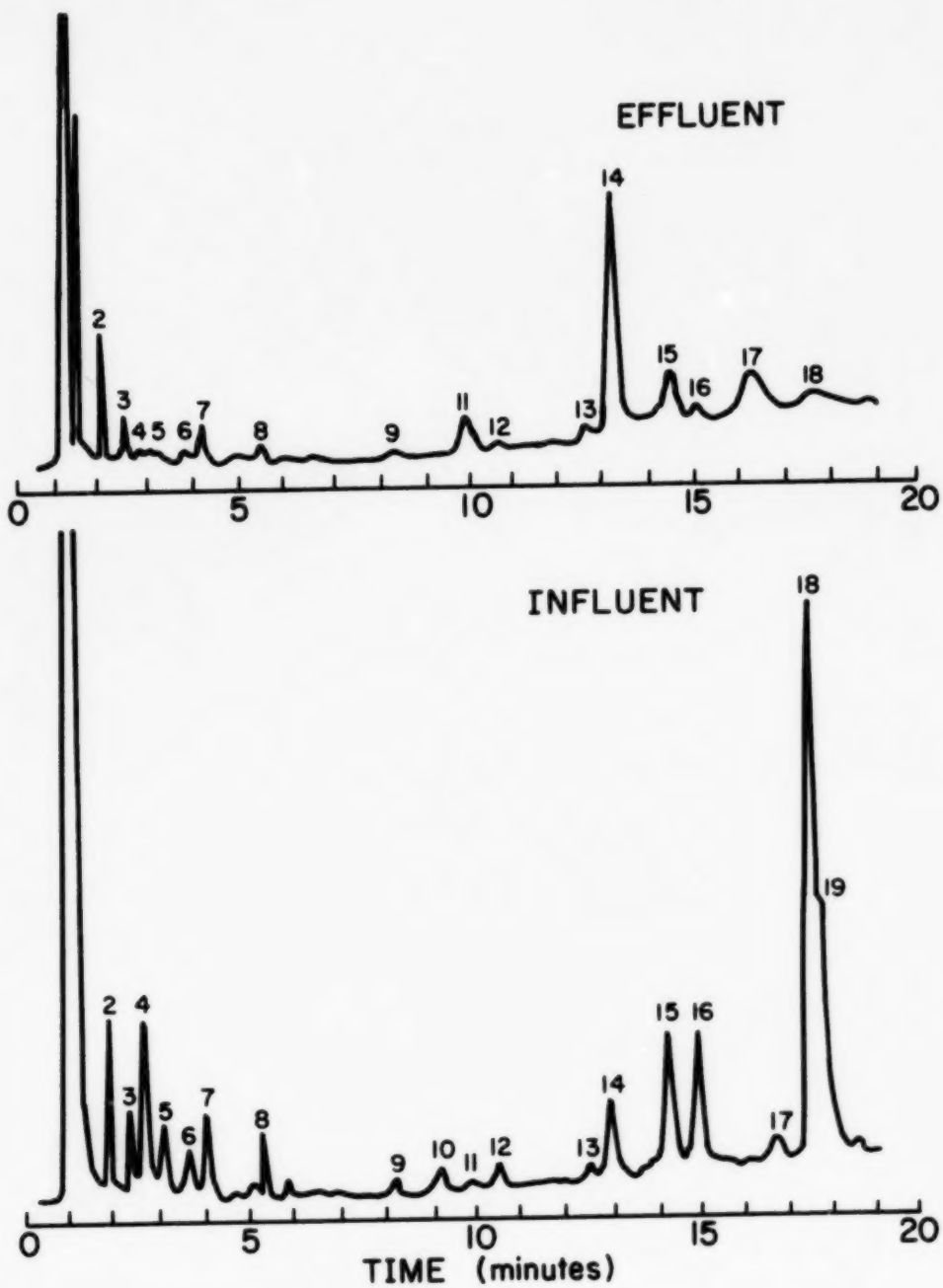

Figure 7. Chromatograms of neutral compounds from red alder, Everett

TABLE 6

NEUTRAL COMPOUNDS FROM THE EVERETT LAGOON - DOUGLAS FIR

Peak No.	Compound Identity	Molecular Weight	M.S. Major Fragments and Comments						Lit. Ref.
			57	43	41	71	55	85	
1	Unknown								
2	Unknown								
3	Unknown	146	correlated with M.S. of known and published M.S.						(8)
4	α-Pinene	136	correlated with published M.S.						(8)
5	Santene	122	correlated with M.S. of known and published M.S.						(8)
6	Camphene	136	✓		✓	✓			(8)
7	Sabinene	136							
8	Unknown								
9	Unknown								
10	β-Pinene	136	correlated with M.S. of known and published M.S.						(8)
11	Unknown	154	91	57	43	41	50	71 (terpene alcohol)	(8)
12	Myrcene	136	correlated with M.S. of known and published M.S.						(8)
13	α-Phellandrene	136	✓		✓	✓			
14	1-4 Cineole		✓		✓	✓			
15	Limonene	136	✓		✓	✓			
16	1-8-Cineole		✓		✓	✓			
17	Unknown								
18	Δ^3-Carene	136	correlated with M.S. of known and published M.S.						(8)
19	p-Cymene	134	✓		✓	✓	✓		
20	Unknown								
21	Terpinolene	136	correlated with M.S. of known and published M.S.						(8)
22	Unknown								
23	Unknown								
24	Unknown								
25	Unknown								
26	Unknown								
27	Fenchone	152	correlated with M.S. of known and published M.S.						(9)
28	Unknown								
29	Unknown	190	154	43	59	81	131	111	
30	Unknown								
31	Unknown								

TABLE 6 (Continued)

NEUTRAL COMPOUNDS FROM THE EVERETT LAGOON - DOUGLAS FIR

Peak No.	Compound Identity	Molecular Weight	M.S. Major Fragments and Comments						Lit. Ref.
			57	43	41	71	55	85	
32	Unknown	124	104	81	108	109	93	95	
33	Unknown								
34	Unknown								
35	Camphor	152	correlated with M.S. of known and published M.S.						(9)
36	Linalool	154	correlated with M.S. of known and published M.S.						(10)
37	Unknown	148	95	67	43	138	123	41	
38	Unknown	148	41	81	95	67	55	69	
39	Fenchyl alcohol	154	correlated with M.S. of known and published M.S.						(11)
40	Unknown								
41	Unknown	148	93	41	91	55	121	39	
42	Terpinene-4-ol	154	correlated with M.S. of known and published M.S.						(11)
43	Unknown								
44	Unknown								
45	α-Terpineol	154	correlated with M.S. of known and published M.S.						(11)
46	Unknown								
47A	Sesquiterpene	204	93	41	91	69	55	43	
47B	Sesquiterpene	204	119	91	134	65	41	39	
48A	Sesquiterpene	204	91	119	93	79	41	55	
48B	Sesquiterpene	204	160	119	91	43	41	105	
49	Anethole								
50	Unknown								
51	Unknown		93	136	91	130	117	77	
52	Unknown		41	67	91	119	57	55	
53	Unknown								

TABLE 7

NEUTRAL COMPOUNDS FROM THE EVERETT LAGOON - WESTERN RED CEDAR

Peak No.	Compound Identity	Molecular Weight	M.S. Major Fragments and Comments	Lit. Ref.
1	Unknown			
2	Unknown		83 85 44 41 47	
3	α-Pinene	136	correlated with M.S. of known and published M.S.	(8)
4	Camphene	136	✓ ✓ ✓	(8)
5	β-Pinene	136	✓ ✓ ✓	(8)
6	Myrcene	136	✓ ✓ ✓	(8)
7	α-Phellandrene	136	correlated with published M.S.	(8)
8	1-4-Cineole	154	correlated with M.S. of known	
9	1-8-Cineole	154	✓ ✓ ✓	
10	Unknown			
11	Fenchone	152	correlated with M.S. of known and published M.S.	(9)
12	Unknown	153	43 153 71 41 81 55	
13	Guaiacol	122	correlated with M.S. of known	
14	Camphor	152	correlated with M.S. of known and published M.S.	(9)
	Tropolone	164	123 138 41 55 67 39 (tropolone)	(12)
15	Tropolone	164	95 41 123 55 67 119 ✓	(12)
16	Fenchyl alcohol	154	correlated with M.S. of known and published M.S.	(11)
17	Tropolone	164	93 77 121 79 95 107 (tropolone)	(12)
18	Unknown	164	93 107 79 91 121 41 ✓	(12)
19	α-Terpineol	154	correlated with M.S. of known and published M.S.	(11)
20	Unknown	206	119 93 41 91 76 79	

TABLE 8

NEUTRAL COMPOUNDS FROM THE EVERETT LAGOON, RED ALDER

Peak No.	Compound Identity	Molecular Weight	M.S. Major Fragments and Comments						Lit. Ref.
			43	57	41	71	93	85	
1	Unknown	142							
2	Acyclic Terpene								
3	Unknown	148	54	43	41	71	44	55	
4	α-Pinene	136	correlated with M.S. of known and published M.S.						(8)
5	Camphene	136	✓		✓	✓		✓	(8)
6	β-Pinene	136	✓		✓	✓		✓	(8)
7	Myrcene	136	✓		✓	✓		✓	(8)
7A	1-4 Cineole		correlated with M.S. of known						
7B	Limonene	136	correlated with M.S. of known and published M.S.						(8)
8	1-8-Cineole		correlated with M.S. of known						
8A	β-Phellandrene	136	correlated with M.S. of known						
9	Unknown								
10	Unknown								
11	Fenchone	152	correlated with M.S. of known and published M.S.						(9)
12	Unknown	153	43	46	153	71	81	55	
13	Unknown								
14	Camphor	152	correlated with M.S. of known and published M.S.						(9)
15	Fenchyl alcohol	154	✓			✓	✓		(11)
16	Terpinene-4-ol	154	✓			✓	✓		(11)
17	Unknown								
18	α-Terpineol	154	correlated with M.S. of known and published M.S.						(11)
19	Unknown								
20	Unknown	138	94	41	43	55	44	57	

TABLE 9

CONCENTRATION OF NEUTRAL COMPOUNDS ENTERING AND LEAVING THE EVERETT AERATED LAGOON, DOUGLAS FIR

(ppm)

GC peak[a]	Compound Identification	ABI 12-21[b]	ABE 12-21[c]	GC peak	Compound Identification	ABI 12-12	ABE 12-21
1	*		.02	34	*	.04	
2	*		.07	35	Camphor	.06	.03
3	*	.02	.02	36	Linalool		tr
4	α-Pinene	.14	.04	37	*	.02	tr
5	Santene	.01	.05	38	*	.02	tr
6	Camphene	tr		39	Fenchyl alcohol	.10	
7	Sabinene	tr	.03	40	*		tr
8	*		.03	41	*	.03	
9	*		.01	42	Terpinene-4-ol	.12	
10	β-Pinene	.01	.04	43	*		tr
11		tr	.02	44	*		.01
12	Myrcene	tr		45	α-Terpineol	.39	
13	α-Phellandrene	tr		46	*	.33	
14	1-4-Cineole	.01	.01	47	Sesquiterpene	.01	.01
15	Limonene	.01	.02	48	Sesquiterpene	.02	.01
16	1-8-Cineole	.01	.01	49	Anethole	.02	.01
17	*	.01	.01	50	*	tr	tr
18	Δ^3-Carene	tr	tr	51	*	.06	.03
19	p-Cymene	.01	tr	52	*	.06	.04
20	*	tr		53	*	.03	.03
21	Terpinolene	.01	.01				
22	*		.01				
23	*		.02				
24	*	.01					
25	*	.02	.01				
27	Fenchone		.02				
29	*	.07	tr				
30	*		.01				
31	*		.02				
32	*	.01			TOTAL	1.85	.65

(a) Keyed to Figure 5
(b) Aeration Basin Influent and Effluent
(c) Sample date
(*) Unknown

TABLE 10

CONCENTRATION OF NEUTRAL COMPOUNDS ENTERING AND LEAVING THE EVERETT AERATED LAGOON, ALDER AND CEDAR

(ppm)

GC peak[a]	Alder Compound Identification	ABI 2-26	ABE 3-5	GC peak	Cedar Compound Identification	ABI 2-28	ABE 3-7
2	Acyclic terpene	.05	.04	1		.05	.03
3		.03	.02	2		.01	.01
4	α-Pinene	.08	tr	3	α-Pinene	.01	.06
5	Camphene	.02	tr	4	Camphene	tr	tr
6	β-Pinene	.02	tr	5	β-Pinene	tr	.02
7	Myrcene	.03	.01	6	Myrcene	.03	.02
7A	1-4-Cineole	tr	tr	7	α-Phellandrene		.01
7B	Limonene	tr		8	1-4-Cineole	.02	tr
8	1-8-Cineole	.02	.01	9	1-8-Cineole	.02	.01
8A	β-Phellandrene	tr		10		.01	
9		.01	tr	11	Fenchone		.04
10		.01		12		.05	tr
11	Fenchone	tr	.03	13	Guaiacol	tr	tr
12		.02	tr	14	Tropolone & Camphor	.69	
13		.01	tr	14A	Camphor		.31
14	Camphor	.05	.23	15	Tropolone	.13	tr
15	Fenchyl alcohol	.11	.03	16	Fenchyl alcohol		.08
16	Terpinene-4-ol	.08		17	Tropolone	.23	.02
16A			.06	18		.39	
17		.01		19	α-Terpineol	.26	tr
18 & 19	α-Terpineol	.65	tr	20		.02	.03
	TOTAL	1.20	.42		TOTAL	1.92	.61

(a) Keyed to Figures 6 and 7

a different GC column and at different sensitivity. These results are not surprising since Douglas fir is the only one of these species that contains any terpenes. The results point out that some mixing of wood species probably occurs, and certainly water reuse and recirculation is occurring to a great extent so that species changes for a few days has little influence on the mill effluent organics.

The concentration of terpenes entering the Everett lagoon is only 15-20% of that found in the Springfield lagoon. This is mainly due to simple dilution because of the bleach plant effluent and also partially due to differences in the type of processing equipment, i.e., the use of batch digesters at Everett and of a continuous digester at Springfield. The treatment efficiency of the Everett lagoon averages about 65% removal of these compounds while Springfield removes 88%.

SECTION VIII

ACIDIC COMPOUNDS

The acidic compounds isolated in this fraction are a variety of resin and fatty acids, neutral terpenes and some phenolics. Most of these occur as such in the wood being pulped, although some composition changes take place in the resin acids, and the phenols are formed in the digester. The fraction is very complex because the method of isolation tends to collect all acidic and neutral compounds of low solubility in water, including neutral terpenes as found in the preceding section. These compounds find their way to the mill effluent in weak wash water, liquor spills and a variety of other streams.

The organic material was isolated from the lagoon samples by adsorption on Amberlite XAD-2 resin[R]. Typically, sample pH was adjusted to 7.0 and 5 gallons were passed through a 1400 ml bed volume column (5 cm x 80 cm) of resin at a flow rate of three bed volumes/hr. The column was washed with four bed volumes of distilled water and the organic material was eluted with five liters of methanol. The methanol and residual water was removed by vacuum rotary evaporation at 30° and the residue taken up in 100 ml of water. This was acidified to pH 3.0 with HCl and extracted with ether (3 x 200 ml). The ether was dried over Na_2SO_4 and concentrated with a Kuderna-Danish evaporator. The compounds were derivatized by forming their respective methyl derivatives by reaction with diazomethane.

Analysis of the derivatized samples was done with a Perkin-Elmer 990 gas chromatograph using a 50 ft. Carbowax 20M-TPA S.C.O.T. column, which was temperature programmed 100-200° at 4°/mon. The injector and manifold temperatures were maintained at 250°C. Column flow was about 6 cc/mm.

Typical gas chromatograms of the compounds isolated from samples of lagoon influent and effluent are shown in Figure 8 and the compounds detected, on which qualitative and quantitative information was sought, are numbered 1 through 38.

[R]Registered Trademark - Rohm and Haas Company

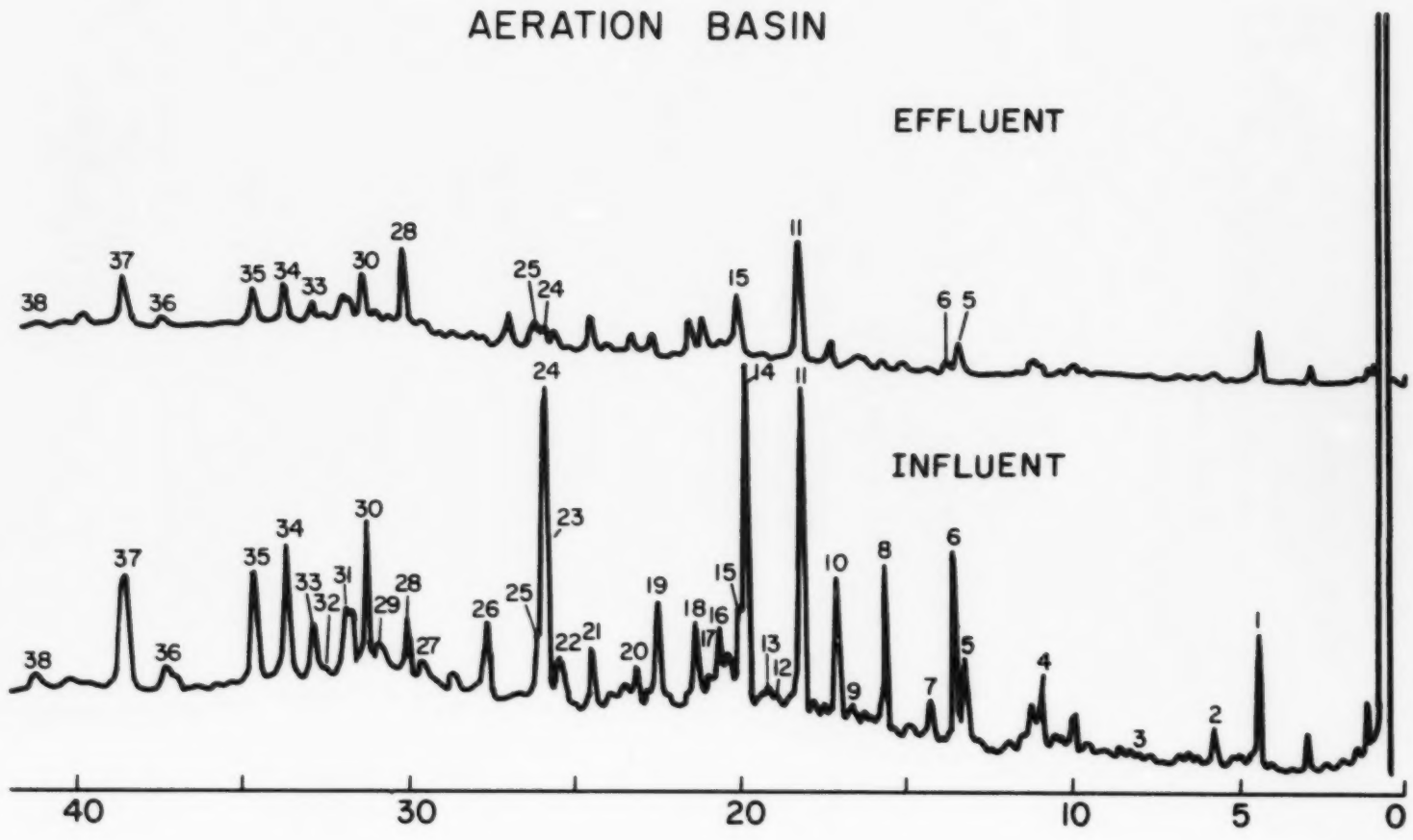

Figure 8. Gas chromatograms of acidic compounds-Springfield

Qualitative identifications were done by determining the mass spectras of the compounds and wherever possible by comparison with spectra obtained from known compounds. Reference was made to spectra reported in the literature also. The data on compound identification is summarized in Table 11. Several resin acids were identified and a number of others were detected but not identified. These are components of the wood resin and are expected in the mill effluent. Also several of the usual fatty acids are found, here perhaps the relatively greater abundance of the saturated acids than unsaturated is surprising. Another acid tentatively identified is p-tolyl-valeric acid, which has recently been reported as a major extractive in Douglas fir. The hydrocarbon abietane also is found and is from this source. Very few phenols were identified, and this was found to be due to extremely poor retention of this class of compounds by the XAD-resin. This was discovered too late to correct for in this study.

Quantitative data was obtained from the GC curves by use of an internal standard technique, a known amount of margaric acid (17:0) was added prior to reaction with diazomethane. Quantitative analysis was facilitated by use of an electronic integrator. Relative response factors for a number of knowns were determined and found to range between 0.96 and 1.02, these were not used in calculations. Quantitation of unidentified peaks was done using a response factor of 1.0.

The recovery efficiency was determined by processing known quantities of individual compounds through the analysis system, and the efficiency was found to be concentration dependent. At concentrations above 1 ppm recovery was about 95% while below this level the efficiency dropped to around 85%, Figure 9.

The quantitative data is summarized in Table 12. The p-tolyl-valeric acid is the major component, sometimes as high as 25 ppm in mill wastewater, and this compound is completely eliminated in the lagoon. The largest concentration of compounds found are the resin

TABLE 11

IDENTIFICATION OF ACIDS

Peak No.	Compound Identity	Molecular Weight	M.S. Major Factors						Comments
1		152	95	81	109	108	83	152	
2		154	93	121	95	91	121	107	
3		136	93	68	121	67	136	79	
4		178	119	91	178	120	117	77	
5									
6		192	119	91	132	117	135	192	
7									
8		146	131	119	146	91	117	115	
9		136	93	68	121				(p-Hydroxybenzaldehyde)
10	p-Tolyl valeric acid	206	119	132	133	91	206	117	
11		220	119	132	145	91	117	220	
12		220	119	132	133	91	175	117	
13									
14	Abietane	272	119	93	105	91	229	272	Hydrocarbon
15	Methyl palmitate		correlated with known						Palmitic acid
16									
17									
18									
19	Veratraldehyde		correlated with known						Vanillin

TABLE 11 (Continued)

IDENTIFICATION OF ACIDS

Peak No.	Compound Identity	Molecular Weight	M.S. Major Factors	Comments
20				
21				
22				
23	Methyl stearate		correlated with known	Stearic acid
24	Methyl oleate		" " "	Oleic acid
25	Methyl linoleate		" " "	Linoleic acid
26				
27				
28				
29		316		(Resin acid)
30		316		(Resin acid)
31	Methyl pimarate		correlated with known	Pimaric acid
32	Methyl sandaracopimarate		correlated with known	Sandaracopimaric acid
33		316	242 301 316	(Resin acid)
34		316		(Resin acid)
35	Methyl isopimarate		correlated with known	Isopimaric acid
36	Methyl abietate		correlated with known	Abietic acid
37	Methyl dehydroabietate		" " "	Dehydroabietic acid
38	Methyl neoabietate		" " "	Neoabietic acid

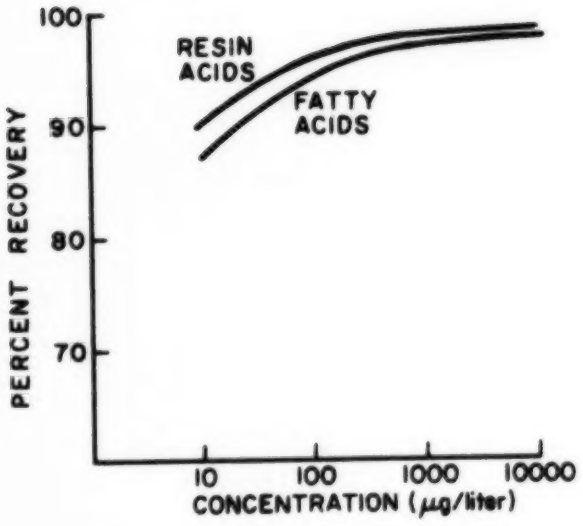

Figure 9. Recovery efficiency of acidic compounds

TABLE 12

CONCENTRATION OF ACIDS ENTERING AND LEAVING THE SPRINGFIELD AERATED LAGOON

(ppb)

Peak No.	ABI 1-31-73	ABE 2-7-73	ABI 2-7-73	ABE 2-14-73	ABI 2-14-73	ABE 2-21-73	ABI 5-5-72	ABE 5-5-72	ABI 9-13-72	ABE 9-21-72
1		35		tr		tr			50	20
2	tr		45		tr		260	tr	tr	
3	tr		tr		560	tr	1300	60	40	
4	720									
5	tr	100		40		20				
6			110	50	tr		80	30	100	tr
7	tr	25	30						30	
8	240	50	130		220				80	
9	520		160		440					
10	2520	tr	170		1660	tr	220		80	tr
11	240	70	170	40			80	tr	180	120
12	480	20	50	tr	320	tr	740	60	tr	
13	780		tr		220		tr			
14	tr	100	340	180	1280	40	320	50	200	60
15	600	40	180	60	1050	tr	140	40	40	
16	tr	30	50				100		50	
17	190	tr	170	tr	740	tr	180	tr	tr	20
18	180	60	60	60	280	40		60	50	20
19	200	tr	170	tr	300	tr	80		60	

TABLE 12 (Continued)

CONCENTRATION OF ACIDS ENTERING AND LEAVING THE SPRINGFIELD AERATED LABOON

Peak No.	ABI 1-31-73	ABE 2-7-73	ABI 2-7-73	ABE 2-14-73	ABI 2-14-73	ABE 2-21-73	ABI 5-5-72	ABE 5-5-72	ABI 9-13-72	ABE 9-21-72
20	tr	20		40	tr		tr		20	
21		10		30	tr	50			30	20
22			90	50	90	tr	tr	30	50	
23	200	160	290	80	360	100	150	tr	300	20
24	120	tr	100	50			tr		70	20
25	120	60	140		240	30	150	40	90	
26	170	25	120	70	340	50	300	40	30	20
27	130	20	130	35	190	140	120	70	50	40
28	180	tr	160		300		80	tr	tr	
29	170	60	150	90	300	30	90	50	140	20
30	160	70	120	60	320	60	120	60	300	80
31	340	150	440	180	1000	140	220	90	240	60
32	tr	tr	100	tr	tr		tr		tr	
33	160	tr	170	tr	440	50	370	40	170	40
34	300	80	580	110	490	70	300	70	380	60
35	330	100	380	50	720	80	340	tr	300	70
36	240	50	470	80	140	tr	290	60	200	30
37	950	120	1030	150	1440	150	880	110	560	120
38	tr		tr		tr		120	tr	tr	20

acids which were found ranging from 4.8 to 2.3 ppm entering the lagoon, averaging 3.2 ppm. The levels in the lagoon discharge were considerably lower, ranging from 0.7 to 0.5 ppm and averaging 0.6 ppm. This is a reduction in concentration of about 80%, fairly comparable to the overall lagoon BOD efficiency which is in the 85 to 87% range usually.

The fatty acids are the next major class of compound found, these ranged from 3.1 to 0.7 ppm entering the lagoon with an average of 1.5 ppm. Again the levels in the lagoon effluent were much lower, ranging from 0.6 to 0.1 ppm and averaging 0.3 ppm, about 80% removal. The saturated fatty acids palmitic and especially stearic seem to be more difficult to remove than the unsaturated acids, this is in agreement with other observations on sewage treatment plant performance.

The information on phenols is disappointing. Early in this study a number of phenols were detected and identified, including guaiacol and syringol, however a change in analysis methods to the resin isolation rather than solvent extraction effectively eliminated the phenols from the recovered compounds and the quantitative data reported in Table 12 on phenols is not reliable.

SECTION IX

POLAR WATER SOLUBLE COMPOUNDS

Polar compounds as used in this report is defined as those organic compounds that cannot be extracted from aqueous solutions by organic solvents. The types of compounds of this class found in kraft pulp mill effluent are mostly polysaccharide degradation products such as sugar acids and their fragmentation products. In the lagoon effluent many products of bacterial metabolism also appear. Most sugar acids remain in the black liquor and are burned in the recovery furnace, however, there is an appreciable amount of these compounds that reaches the lagoon via weak wash water, black liquor spills and other miscellaneous sources, so it was necessary to include this class of compounds in this study.

Lagoon influent and effluent samples were prepared for analysis by filtering, adjusting pH to 7.0, and passing one liter of the sample over a 50 ml column bed of XAD-2 resin at a 2 ml/min flow rate for removal of most of the low solubility neutral and acidic organic materials. The column eluate was then adjusted to pH 8.5 and concentrated 60 fold using a heated rotary evaporator. The pH was raised to 9.2 and sodium tetraborate and methanol added to adjust their concentration to 0.03 M and 2% respectively. This solution was then analyzed by liquid chromatography.

The liquid chromatograph used a 2 liter solvent reservoir, a Waters 3000 pump, a septum injector, a 2.4 mm ID x 50 cm stainless steel column packed with 35-37 micron Bio-Rad ^{R} AGI-X10 anion resin, and a Waters Differential refractometer detector. Data was recorded with a Varian A-25 recorder. Elution was done with a 0.03 M sodium tetraborate 2% methanol solvent.

Compound identifications were made by collecting eluted portions corresponding to detector peaks, preparing trimethylsilyl (TMS) deriva-

tives, checking these for purity by gas chromatography and then obtaining their mass spectra via the liquid sample inlet system in the mass spectrometer. The scheme of analysis and identification is illustrated in Figure 10.

Individual detector peaks were collected as fractions from the liquid chromatograph, freeze dried and converted directly to the TMS derivatives. Five mg of the acid salt fraction were mixed with 0.4 ml pyridine, 0.2 ml N,O-bis-trimethylsilyl-acetamide and 0.1 ml trimethylchlorosilane. After reaction at room temperature for 4 hours the mixture was rotary evaporated to dryness at 35°C and the residue dissolved in hexane. Compound purity was checked by gas chromatography using a 3% QF-1, 1/8 in. OD by 6 ft. packed column. A temperature program of 70 to 180°C was used so that solvent and any remaining reagents were well separated from the derivatized acid. Examples of the gas chromatographs of several of the TMS derivatives of these acids are shown in Figure 11. If the compound was sufficiently pure, the sample was evaporated to dryness and introduced into the mass spectrometer through either the solid probe or the liquid inlet.

Typical mass spectra obtained by these methods are shown in Figure 12. Examination of these examples shows that there are not large differences in the spectra of the sugar acids, and consequently identification had to be made by running knowns through the same procedure on our mass spectrometer. The methods of identifications are summarized in Table 12, and a typical liquid chromatogram from a lagoon sample with the identified compounds indicated is shown in Figure 13.

The sugar acids are not as well known as most of the other organic compounds encountered in this study and therefore an illustration of the mass spectra of the TMS derivative of glucometasaccharinic acid, including a schematic showing the fragmentation pattern is shown in Figure 14 and the fragment losses resulting in the observed mass spectra

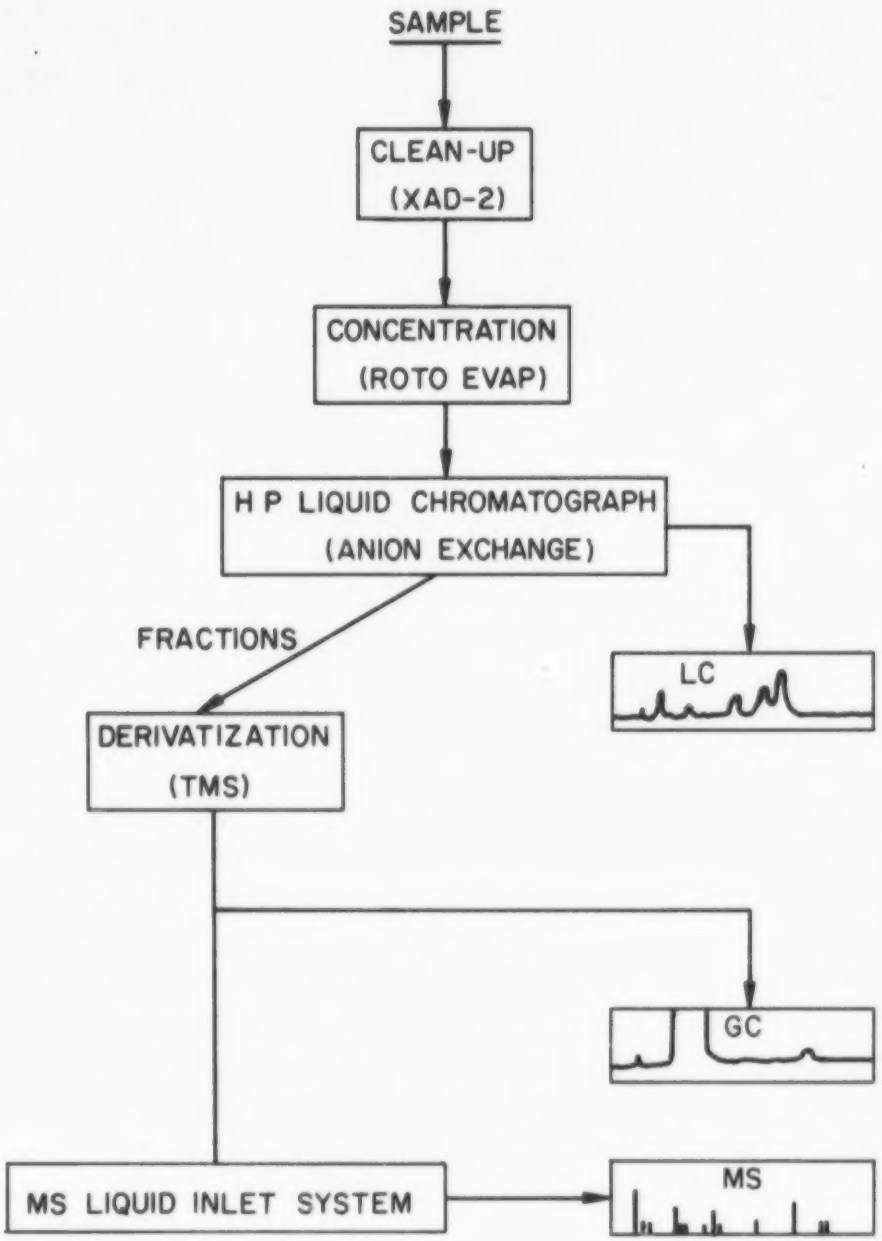

Figure 10. Polar compound analysis and identification

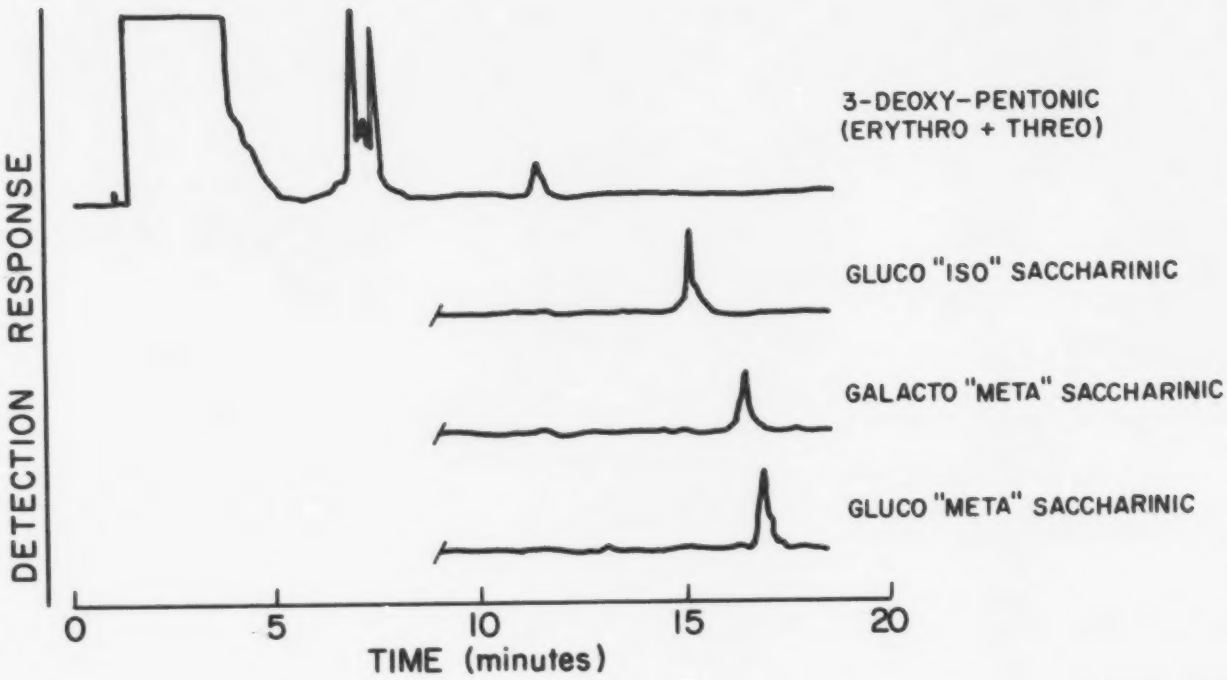

Figure II. Gas chromatograms of trimethylsilyl derivatives of some polar acids

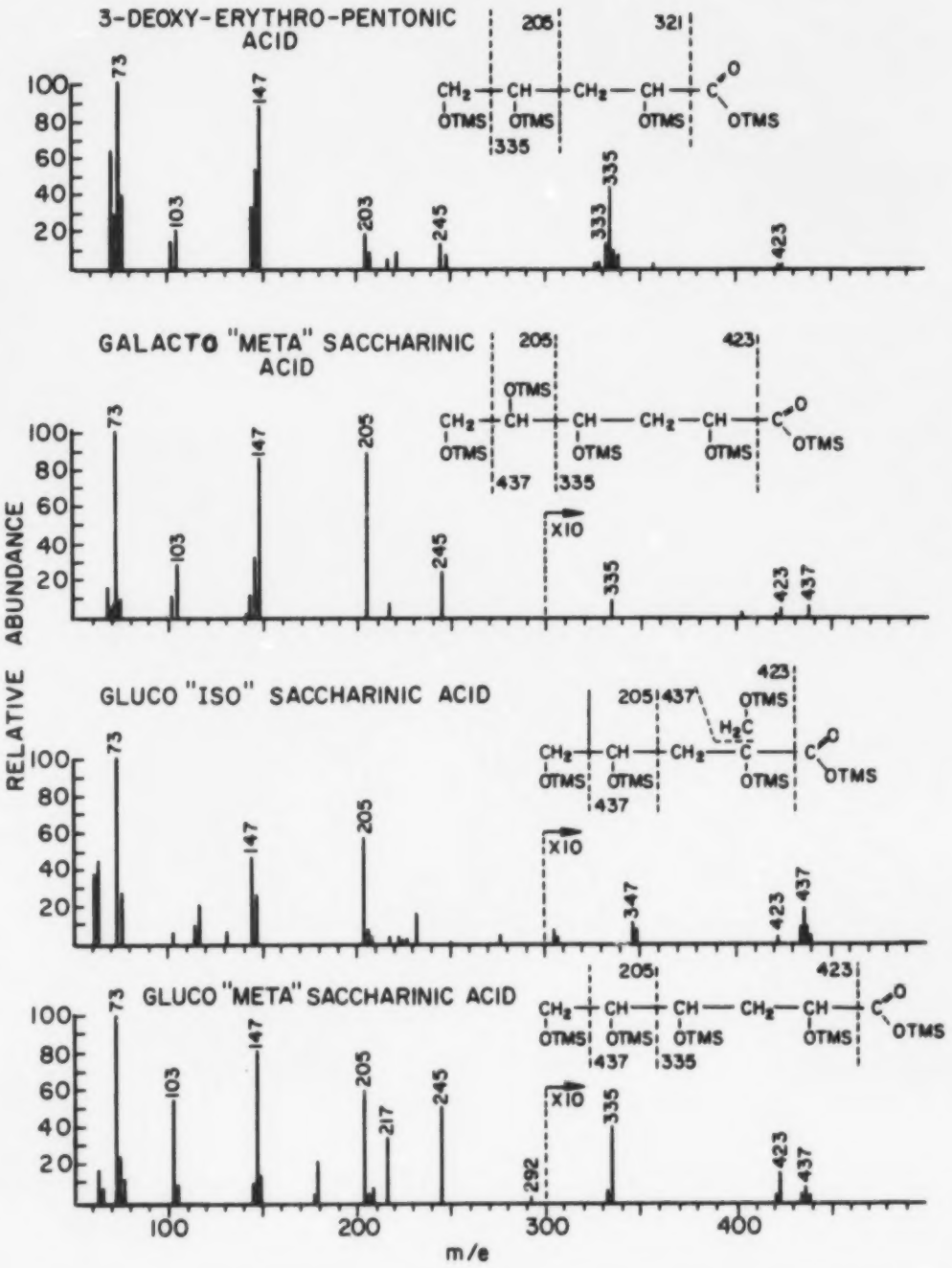

Figure 12. Trimethylsilyl derivatives of some saccharinic acids

TABLE 13

IDENTIFICATION METHODS FOR POLAR COMPOUNDS

Peak No.	Compound Identity	Identification Method	Reference
1	Formic	LC retention correlated with purchased - two different buffers	
2	Acetic	same	
3	Glycolic	same	
4	2-Hydroxybutyric	same	
5	Lactic	same	
6	3-Deoxy-D-threo-pentonic	LC retention correlated with synthesized	
		GC retention correlated with published value	(13,14)
7	3-Deoxy-D-erythro-pentonic	LC retention correlated with synthesized	
		GC retention correlated with published value	(13,15)
		Mass spectra correlated with published spectra	(16)
8 & 9	α& β-D-Gluco"iso"saccharinic	LC retention correlated with synthesized	
		GC retention correlated with published value	(14,15)
		Mass spectra correlated with published spectra	(16)
10	β-D-Gluco"meta"saccharinic	LC retention correlated with published value	(17,18)
		GC retention correlated with published value	(13,15)
		Mass spectra correlated with published spectra	(16)
11	α-D-Gluco"meta"saccharinic	LC retention correlated with published value	(18)
		GC retention correlated with published value	(13,15)
		Mass spectra correlated with published spectra	(16)
12	Unknown		
13	-D-Galacto"meta"saccharinic	GC retention correlated with published value	(13)
		Mass spectra correlated with published spectra	(16)

1 FORMIC ACID
2 ACETIC
3 GLYCOLIC
5 LACTIC
6 3-DEOXY-D-THREO-PENTONIC
7 3-DEOXY-D-ERYTHRO-PENTONIC
8 -D-GLUCO "ISO" SACCHARINIC ACID
9 -D-GLUCO "ISO" SACCHARINIC
10 β-GLUCO "META" SACCHARINIC
11 α-GLUCO "META" SACCHARINIC
12 UNKNOWN
13 -D-GALACTO "META" SACCHARINIC

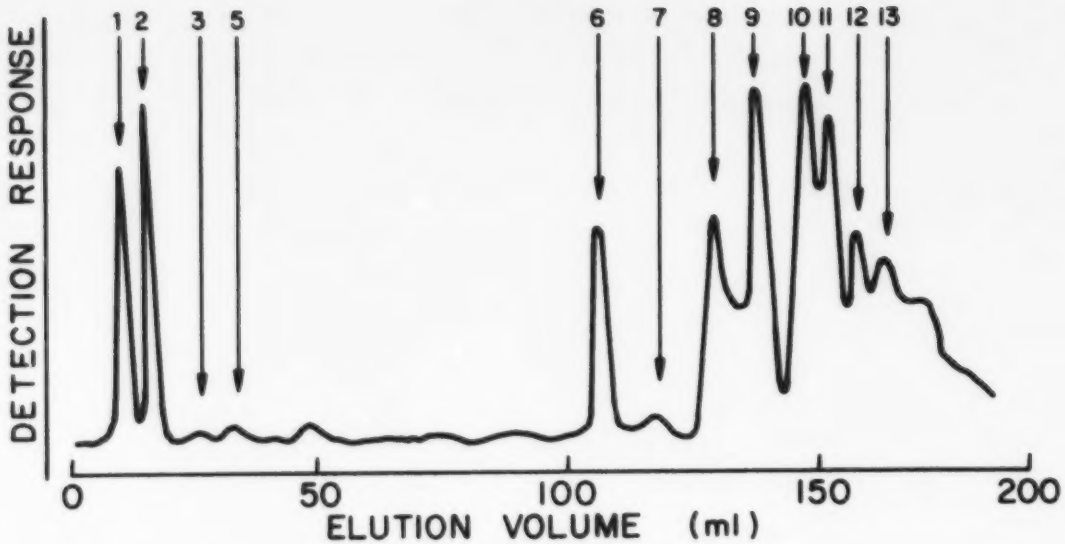

Figure 13. Liquid chromatogram of polar acids - lagoon influent 2/14/73

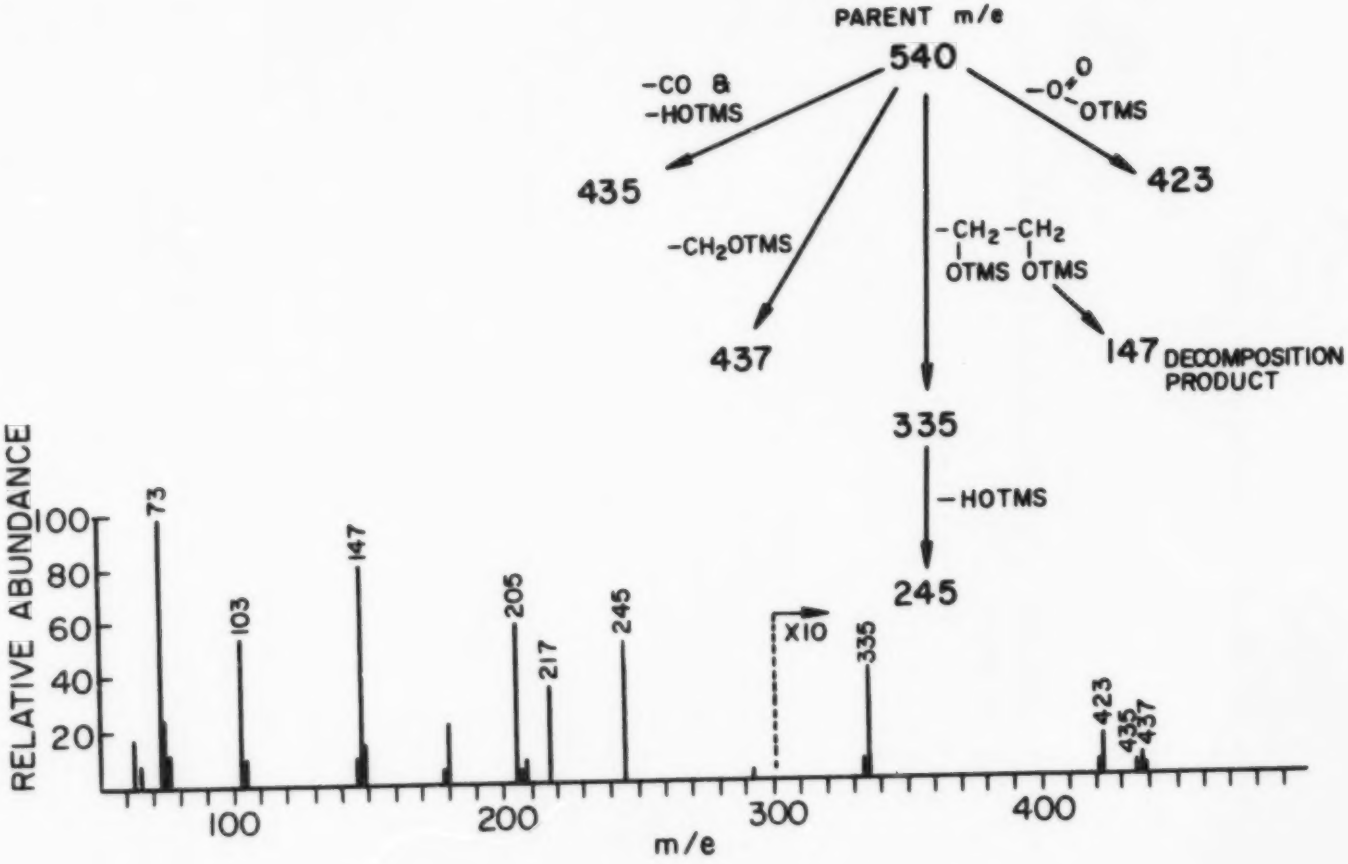

Figure 14. Mass spectra fragmentations - TMS derivative of glucometasaccharine acid

are summarized in Table 14. The primary fragmentations are carbon chain cleavage.

TABLE 14

FRAGMENTATION OF THE TMS DERIVATIVE
OF GLUCOMETASACCHARINIC ACID

m/e(T)	RATIONAL
437 (1)	Loss of C-6 from m/e 540 parent
435 (1)	Loss of carbon monoxide and trimethylsilanol from m/e 540 parent
423 (2)	Loss of C-1 from m/e 540 parent
335 (4)	Loss of C-5 and -6 from m/e 540 parent
245(45)	Loss of trimethylsilanol from m/e 335
217(17)	Loss of carbon monoxide from m/e 245
205(55)	C-5 and -6 TMS fragment
147(80)	Decomposition from m/e 205
103(55)	C-6 TMS fragment Also a rearrangement product
73(100)	Trimethylsilyl ion (base peak)

The polar compounds identified in lagoon samples are essentially the saccharinic acids and further degradation products expected from alkaline treatment of polysaccharides. A number of small molecule organic acids such as acetic are observed which may also arise as a result of metabolic processes occurring in the lagoon.

Quantitative data was obtained from the liquid chromatograph by calibration with purchased or synthesized known compounds. Unavailable saccharinic acids (galacto) were quantitatively correlated by dichromate oxidation of individual fractions with the other knowns. Data obtained on the Springfield and Everett lagoons is summarized in Table 15 which shows concentrations of the individual acids in ppm, and Table 16

TABLE 15

CONCENTRATION OF POLAR ORGANIC ACIDS IN AERATED LAGOONS

(ppm)

Peak No.	Compound Identity	Springfield ABI 7/17/72	ABE 7/25	ABI 9/13	ABE 9/21	ABI 2/14/73	ABE 2/21	ABI 5/10	ABE 5/17	Everett ABI 2/28/73
1	Formic	15.6	35.2	11.2	77.5	36.0	6.5	9.6	5.5	17.2
2	Acetic	35.2	29.2	13.5	7.2	56.0	3.0	10.2	6.0	9.6
3	Glycolic	2.4	1.0	2.0		2.5	tr	2.1	0.7	3.8
4	2-Hydroxy butyric	1.2	0.8	2.0			tr	2.8	0.4	2.0
5	Lactic	3.3	0.9	2.0	1.5	5.0	tr	1.3	0.5	4.0
6	3-Deoxy-D-threo-pentonic	0.8	0.4	4.5		32.5	nd	nd	nd	8.8
7	3-Deoxy-D-erythro-pentonic	0.6		6.5		3.0	nd	nd	nd	1.3
8	α or β-D-Glucoiso saccharinic					37.0	nd	nd	nd	1.4
9	α or β-D-Glucoiso saccharinic					58.0	nd	nd	nd	3.8
10	β-D-Glucometa saccharinic	14.4	2.0	79.5	1.0	60.0	nd	nd	nd	4.4
11	α-D-Glucometa saccharinic					53.0	nd	nd	nd	5.2
12	Unknown	8.8	0.8	22.0	tr	32.0	nd	nd	nd	5.7
13	D-Galactometa saccharinic	2.8	0.4	21.0	3.5	29.0	nd	nd	nd	2.8
14	Other Acids	11.8	13.7	9.0	7.0	30.0	18.0	16.7	10.0	41.8
	TOTAL	96	84	173	97	436	27	43	23	112

TABLE 16

TREATMENT EFFICIENCY FOR POLAR ORGANIC ACIDS

	TOTAL IN PPM PEAKS 1 - 13	TOTAL IN PPM ALL PEAKS
SUMMER		
07-18-72 IN	84.6	96.4
07-25-72 OUT	70.7	84.4
QUANTITY REMOVED	13.9	12.0
PERCENT REMOVED	16.6%	12.4%
AUTUMN		
09-13-72 IN	164.2	173.2
90-21-72 OUT	90.7	97.7
QUANTITY REMOVED	73.5	75.5
PERCENT REMOVED	44.8%	43.6%
WINTER		
02-14-73 IN	406.0	436.0
02-21-73 OUT	9.5	27.5
QUANTITY REMOVED	396.5	408.5
PERCENT REMOVED	97.7%	93.7%
SPRING		
05-10-73 IN	26.0	42.7
05-17-73 OUT	13.1	23.1
QUANTITY REMOVED	12.9	19.6
PERCENT REMOVED	49.6%	45.9%

which presents a summary of the seasonal treatment efficiency of these compounds.

It seems appropriate to discuss these acids in two categories; the sugar acids which arise from the pulp mill only, and the small molecule acids such as acetic and formic, which are also metabolic products formed in the lagoon.

The lagoon is very effective at all times in eliminating the sugar acids from the wastewater stream. The case with formic and acetic acid is much different. Acetic acid is lowered in concentration across the lagoon an average of 60%, and in the best case (Winter 1973) over 94%. Formic acid, however, is actually produced by the lagoon. This effect is thought to be a function of the operating pH, since metabolism of formic acid is known to be retarded at pH 7.4 and above (19). Strained oxygen conditions within the lagoon also interfere with formic metabolism.

Reviewing one years lagoon performance, during the summer 1972 the saccharinic acids are effectively metabolized (80%+), while the acetate and other small molecule acids remains high and formate more than doubles in concentration. The fall 1972 samples showed better overall performance with the saccharinic acids being over 96% removed. Again acetate and other small molecules remain somewhat high, and formate increased seven fold across the lagoon. The winter 1973 samples were taken during a heavier loading period, probably some liquor spill was involved here as indicated by the unusually large amount of saccharinic acids being fed. The effluent sample corresponding to this influent was exceptionally clean, showing only traces of acetate and other small molecule acids. The spring 1973 sample, in contrast to the winter sample, shows almost no evidence of liquor in the input, and low output levels.

These results illustrate several things. First, the input levels

to the aerated lagoon vary widely. Even in the limited number of samples taken in this study a 10 fold range can be seen. Secondly, regardless of whatever else is occurring, the sugar derived saccharinic acids are essentially completely removed from the wastewater. Thirdly, acetate, formate and other small molecule acids which are metabolic products in the lagoon are often not significantly lowered in concentration by the lagoon treatment.

SECTION X

CONTROL OF ORGANIC COMPOUNDS IN MILL EFFLUENT

This study has identified simple organic acids, saccharinic acids and related products, resin acids, fatty acids, phenols and various terpenes in the effluent from kraft pulp mills. It has also shown that the aerated lagoon is quite effective in lowering the amounts of these materials so that the treated water discharged from the lagoon contains less than 1 ppm of each class of compound except the simple organic acids, which may be near the 100 ppm range, depending on lagoon operation.

Based on review of the literature, these discharge levels do not seem to be of importance environmentally, with the possible exception of the resin and fatty acids where a wider safety margin would be desirable (20,21). There are recent reports of a variety of bacteria which readily metabolize resin acids (22). At present there does not seem to be much effort to optimize lagoon performance by utilization of specific bacteria or other means and in-plant control has therefore been examined. These acids are non-volatile and can only enter the mill wastewater in an aqueous stream. Some sources of loss are liquor spills, entrainment in the evaporators, weak wash water and bleach plant effluent which is not reused etc. A special and important case is foaming over of equipment. It is known that these acids are associated with foam stabilization and they can be recovered from black liquor by foam fractionation. Any process unit which foams over, resulting in a spill, is selectively transferring these acids out of the recovery process and into the wastewater discharge stream. Control of foaming is therefore an essential part of in-process control of discharge of the resin acids. Probably the best way to protect against loss of this type is to provide a system for spill containment which will allow return for recovery of the material. Controlled release to the treatment system is an alternative.

This study shows that the major terpene in the discharged wastewater is α-terpineol. The monoterpenes in the wastewater are about 90% oxygenated compounds and most of this is α-terpineol. This turpentine content is very different from the crude sulfate turpentine from the same mill, which is about 15% oxygenated terpenes. The main reason for this difference is that most of the hydrocarbons are removed in the turpentine collection system, and only the less volatile more water soluble terpenes remain in process water to enter the wastewater streams (23). The main sources are the turpentine decanter underflow, 4500 ppm turpentine and hot water accumulates water, 650 ppm turpentine (24). If further in-plant control is desired, steam stripping of these two streams would lower the turpentine in mill effluent by about one-half and this should help lower the overall terpene discharge.

SECTION XI

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SECTION XII

PUBLICATIONS AND PATENTS

The following publications have been produced or are anticipated to be produced as a result of this project:

1) Wilson, Donald F. and Bjorn F. Hrutfiord, "The Fate of Turpentine in Kraft Mill Aerated Lagoons," presented at the 1974 CPPA Environmental Conference, Toronto. In press, Pulp and Paper Magazine of Canada.
2) Hrutfiord, B. F., T. S. Friberg, D. F. Wilson and J. R. Wilson, "Organic Compounds in Pulp Mill Lagoon Discharge," presented at the 1975 TAPPI Environmental Conference, Denver. Submitted to TAPPI for publication.
3) Hrutfiord, Bjorn F. and Donald F. Wilson, "Turpentine in Kraft Mill Wastewater" presented at the 1975 CPPA regional Environmental Conference, Jasper.
4) Dissertation: "Monoterpenes: Their Fate and Analysis in Kraft Mill Aerated Lagoons and Kraft Processing" Donald Frederick Wilson. University of Washington, 1974.

SECTION XIII

GLOSSARY

Aerated Lagoon	A pond used for BOD removal in which mill effluent is intensively aerated for seven days or so.
Lagoon Influent	Waste water from the entire mill complex including both the pulp mill and paper mill.
GC/MS	A coupled gas chromatograph-mass spectrometer used for organic compound separation and identification.
XAD Resin	A resin capable of removing organic compounds from water which operates by adsorption by weak interactions.
Neutral Compounds	Organic compounds soluble in organic solvents which do not contain acidic or basis functional groups.
Acidic Compounds	Organic acids, including phenols, resin acids and fatty acids present in kraft effluent.
Polar Compounds	Water soluble organic solvent insoluble compounds, mostly derived from polysaccharide degradation.
Terpenes	C_{10} hydrocarbons, ketones and alcohols which are related to isoprene.
Fatty Acids	C_{16} and C_{18} long chain aliphatic acids.
Resin Acids	C_{20} acids from wood rosins.
Extraction	Immiscible solvent pair partition to selectively remove dissolved materials from one liquid into a second.